Supporting Information Part I for

Cubic Ag_2O nanoparticles incorporated mesoporous silica with large bottle-neck like mesopore for the aerobic oxidative synthesis of disulfide

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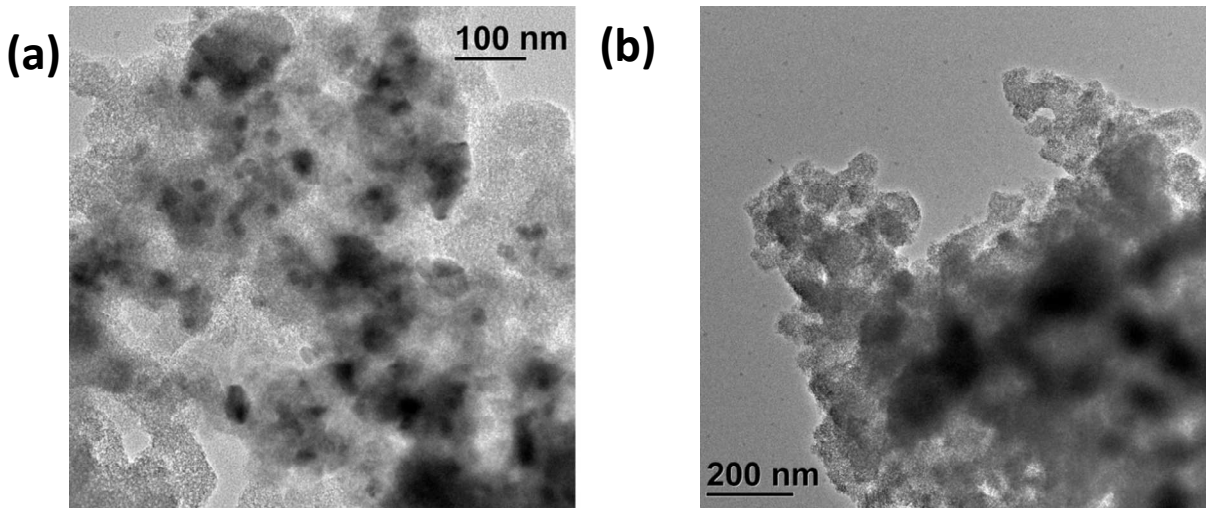


Figure S1. TEM Images of (a) fresh **MSAGN-10** catalyst and (b) five-times reused catalyst.

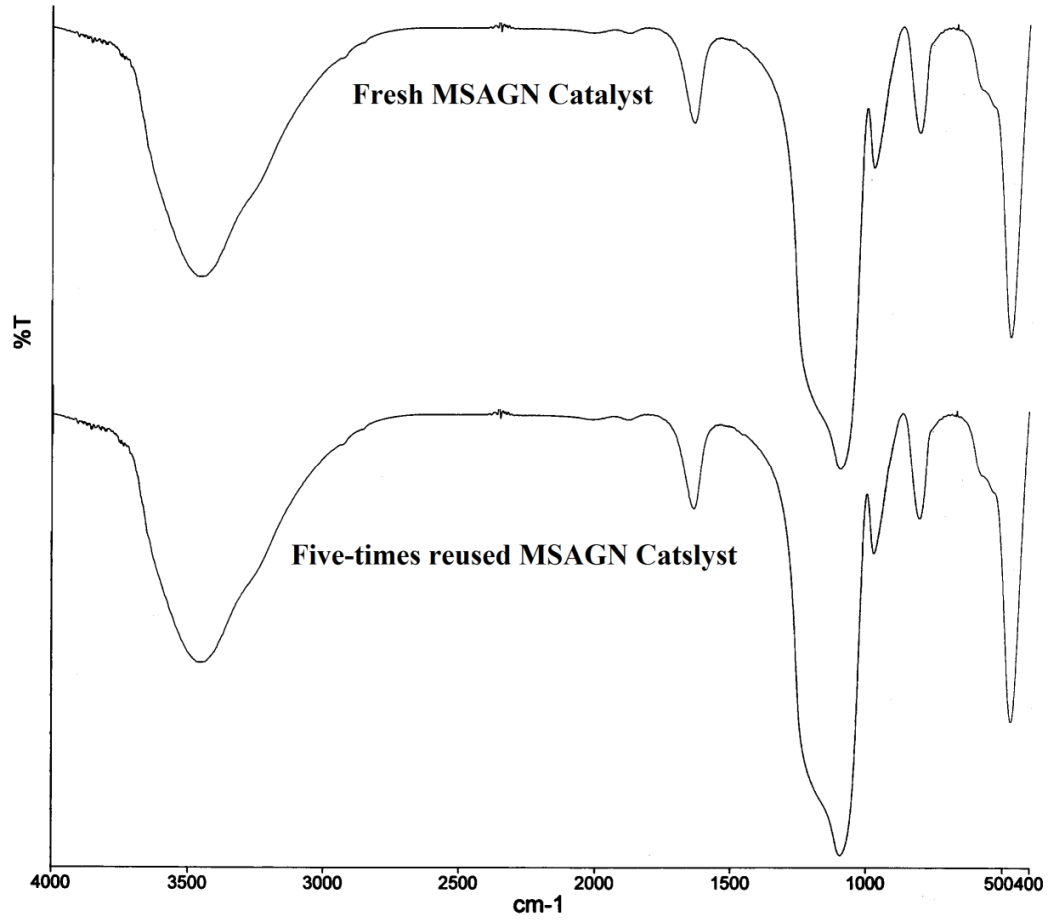
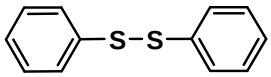


Figure S2. IR spectrum of (a) fresh **MSAGN-10** catalyst and (b) five-times reused catalyst.

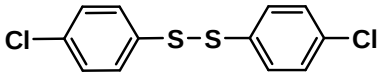
Spectroscopic characterization of 2a-2l

1,2-diphenyldisulfane (2a):



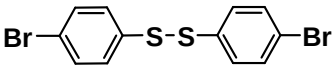
Yield: (0.199g, 91%); White crystalline solid; mp 58−60 °C; IR (KBr) 2379, 2347, 1948, 1573, 1474, 1436, 1384, 1297, 1154, 1071, 1021, 736, 686, 462 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.09-7.14 (m, (2×1)H), 7.17-7.22 (m, (2×2)H), 7.40 (d, *J*=8.1 Hz, (2×2)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 127.1, 127.4, 127.5, 129.0, 129.1, 137.0; Anal. Calcd for $C_{12}H_{10}S_2$: C, 66.01; H, 4.62%. Found: C, 66.03; H, 4.64%. HRMS (m/z) Calcd for $[M+H]^+$: 219.0302. Found 219.0941.

1,2-bis(4-chlorophenyl)disulfane (2b):



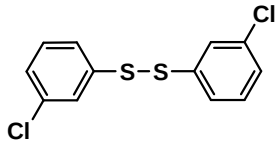
Yield: (0.259g, 90%); White solid; mp 69−71 °C; IR (KBr) 3081, 2361, 2347, 2341, 2291, 1470, 1385, 1100, 1093, 1010, 815, 670, 489 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.16 (dd, *J*=6.75 Hz, *J*=1.8 Hz, (2×2)H), 7.29 (dd, *J*=6.6 Hz, *J*=1.8 Hz, (2×2)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 129.26, 129.3, 133.6, 135.1; Anal. Calcd for $C_{12}H_8Cl_2S_2$: C, 50.18; H, 2.81%. Found: C, 50.20; H, 2.83%.

1,2-bis(4-bromophenyl)disulfane (2c):



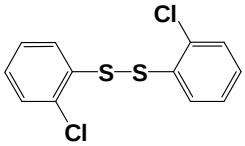
Yield: (0.335g, 89%); White solid; mp 90−92 °C; IR (KBr) 3091, 2369, 2349, 2361, 2281, 1460, 1395, 1109, 1095, 813, 680, 479 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.27 (dd, *J*=7.5 Hz, *J*=1.8 Hz, (2×2)H), 7.35 (dd, *J*=7.8 Hz, *J*=1.8 Hz, (2×2)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 121.6, 129.4, 132.2, 135.7; Anal. Calcd for $C_{12}H_8Br_2S_2$: C, 38.32; H, 2.14%. Found: C, 38.34; H, 2.15%.

1,2-bis(3-chlorophenyl)disulfane (2d):



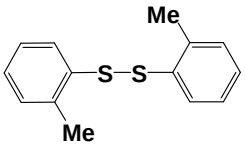
Yield: (0.258g, 90%); White solid; mp 81−83 °C; IR (KBr) 3015, 2399, 2357, 1948, 1574, 1476, 1437, 1364, 1298, 1153, 1071, 1021, 736, 686, 472 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.12-7.20 (m, (2×2)H), 7.26-7.33 (m, (2×1)H), 7.40-7.43 (m, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 125.3, 127.0, 127.5, 130.1, 135.0, 138.4; Anal. Calcd for $C_{12}H_8Cl_2S_2$: C, 50.18; H, 2.81%. Found: C, 50.21; H, 2.81%.

1,2-bis(2-chlorophenyl)disulfane (2e):



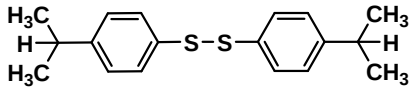
Yield: (0.256g, 89%); White solid; mp 86−88 °C; IR (KBr) 3072, 2366, 2348, 2345, 2299, 1470, 1386, 1101, 1095, 1010, 812, 673, 469 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.02-7.14 (m, (2×2)H), 7.26 (dd, *J*=7.65 Hz, *J*=1.5 Hz, (2×1)H), 7.45 (dd, *J*=7.8 Hz, *J*=1.5 Hz, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 127.3, 127.5, 127.8, 129.7, 131.9, 134.4; Anal. Calcd for $C_{12}H_8Cl_2S_2$: C, 50.18; H, 2.81%. Found: C, 50.19; H, 2.83%.

1,2-di*o*-tolyldisulfane (2f):



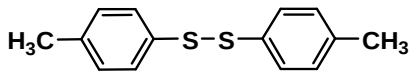
Yield: (0.222g, 90%); White solid; mp 43−45 °C; IR (KBr) 3077, 2963, 2937, 1887, 1483, 1470, 1409, 1193, 1144, 1016, 820, 540, 469 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 2.37 (s, (2×3)H), 7.06-7.10 (m, (2×3)H), 7.45-7.48 (m, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 20.0, 126.7, 127.4, 128.9, 130.4, 135.5, 137.5; Anal. Calcd for $C_{14}H_{14}S_2$: C, 68.25; H, 5.73%. Found: C, 68.27; H, 5.75%.

1,2-bis(4-isopropylphenyl)disulfane (2g):



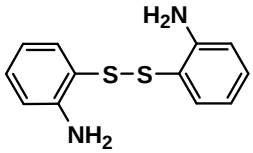
Yield: (0.278g, 92%); White solid; mp 48−50 °C; IR (KBr) 3071, 2960, 2927, 1897, 1493, 1460, 1406, 1183, 1142, 1013, 820, 536, 468 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 1.10 (s, (2×3)H), 1.12 (s, (2×3)H), 2.74-2.78 (m, (2×1)H), 7.04 (dd, *J*=7.8 Hz, *J*=1.8 Hz, (2×2)H), 7.33 (dd, *J*=6.6 Hz, *J*=1.8 Hz, (2×2)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 23.5, 33.4, 126.8, 127.9, 134.0, 147.9; Anal. Calcd for $C_{18}H_{22}S_2$: C, 71.47; H, 7.33%. Found: C, 71.49; H, 7.36%.

1,2-dip-tolyldisulfane (2h):



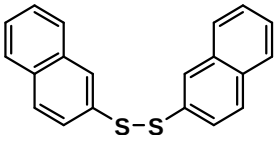
Yield: (0.224g, 91%); White solid; mp 43−45 °C; IR (KBr) 3069, 2970, 2929, 1890, 1495, 1470, 1185, 1132, 1013, 829, 566, 466 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 2.20 (s, (2×3)H), 6.98 (d, *J*=8.1 Hz, (2×2)H), 7.26 (d, *J*=7.2 Hz, (2×2)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 21.0, 128.5, 129.7, 133.9, 137.4; Anal. Calcd for $C_{14}H_{14}S_2$: C, 68.25; H, 5.73%. Found: C, 68.26; H, 5.74%.

2-(2-(2-aminophenyl)disulfanyl)benzenamine (2i):



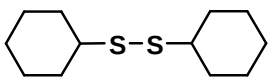
Yield: (0.224g, 90%); Yellow solid; mp 89−91 °C; IR (KBr) 3441, 3375, 3204, 2969, 1677, 1495, 1450, 1356, 1193, 1042, 1003, 726, 486 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 4.16 (br s, (2×2)H), 6.38-6.44 (m, (2×1)H), 6.50-6.54 (m, (2×1)H), 6.95-6.99 (m, (2×2)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 115.1, 118.0, 118.5, 131.4, 136.6, 148.4; Anal. Calcd for $C_{12}H_{12}N_2S_2$: C, 58.03; H, 4.87; N, 11.28%. Found: C, 58.06; H, 4.88; N, 11.30%.

1-(naphthalen-2-yl)-2-(naphthalen-3-yl)disulfane (2j):



Yield: (0.287g, 90%); White solid; mp 143−144 °C; IR (KBr) 3009, 1634, 1559, 1488, 1386, 1136, 1019, 867, 756, 568, 495 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.34-7.41 (m, (2×2)H), 7.55 (dd, *J*=8.55 Hz, *J*=1.8 Hz, (2×1)H), 7.57-7.72 (m, (2×3)H), 7.91 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 125.7, 126.2, 126.6, 126.7, 127.5, 127.8, 129.0, 132.5, 133.5, 134.3; Anal. Calcd for $C_{20}H_{14}S_2$: C, 75.43; H, 4.43%. Found: C, 75.44; H, 4.45%. HRMS (m/z) Calcd for $C_{20}H_{14}S_2$: 318.3537. Found 317.9394.

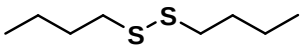
1,2-dicyclohexyldisulfane (2k):



Yield: (0.203g, 88%); Colorless oil; mp oil; IR (KBr) 2969, 2677, 1498, 1430, 1366, 1200, 1163, 1024, 1013, 826, 727, 516, 490 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 1.27-1.34 (m, (2×5)H), 1.62 (br s, (2×1)H), 1.79 (br s, (2×2)H), 2.03 (br s, (2×2)H), 2.68 (t, *J*=3.9 Hz, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$;

Me_4Si) δ 25.6, 26.0, 32.8, 49.8; Anal. Calcd for $C_{12}H_{22}S_2$: C, 62.55; H, 9.62%. Found: C, 62.57; H, 9.64%.

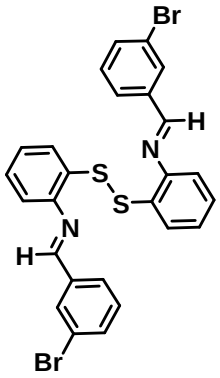
1-Butyldisulfanyl-butane (2l):



Yield: (0.152 g, 85%); Colorless oil; mp oil; IR (KBr) 2989, 2923, 2846, 1488, 1439, 1383, 1240, 1123, 1004, 994, 856, 757, 506, cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 0.86 (t, *J*=7.2 Hz, (2×3)H), 1.22-1.34 (m, (2×2)H), 1.54-1.66 (m, (2×2)H), 2.62 (t, *J*=6.9 Hz, (2×2)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 12.2, 23.4, 32.4, 36.9; Anal. Calcd for $C_8H_{18}S_2$: C, 53.87; H, 10.17%. Found: C, 53.88; H, 10.19%.

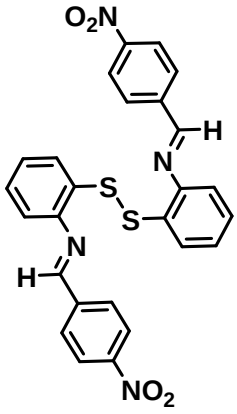
Spectroscopic characterization of 4a-4l

(15*E*)-*N*-(3-bromobenzylidene)-2-(2-((*E*)-2-(3-bromobenzylideneamino)phenyl)disulfanyl)benzenamine (4a):



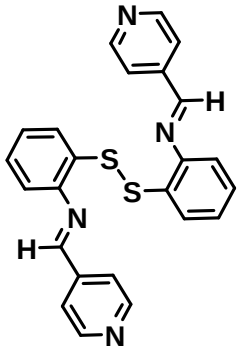
Yield: (0.530g, 91%); Grey colored solid; mp 120−122 °C; IR (KBr) 1632, 1569, 1472, 1467, 1429, 1300, 1229, 1160, 1130, 984, 732, 720, 549, 488 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.05 (d, *J*=0.9 Hz, (2×1)H), 7.07-7.28 (m, (2×2)H), 7.38 (t, *J*=7.5 Hz, (2×1)H), 7.65 (d, *J*=7.2 Hz, (2×1)H), 7.71 (d, *J*=7.8 Hz, (2×1)H), 7.90 (d, *J*=7.5 Hz, (2×1)H), 8.16 (s, (2×1)H), 8.43 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 117.2, 123.0, 126.3, 127.1, 127.2, 127.8, 130.3, 131.6, 132.2, 134.5, 137.9, 148.4, 158.2; Anal. Calcd for $C_{26}H_{18}Br_2N_2S_2$: C, 53.62; H, 3.12; N, 4.81%. Found: C, 53.63; H, 3.13; N, 4.83%.

(15*E*)-*N*-(4-nitrobenzylidene)-2-(2-((*E*)-2-(4-nitrobenzylideneamino)phenyl)disulfanyl)benzenamine (4b):



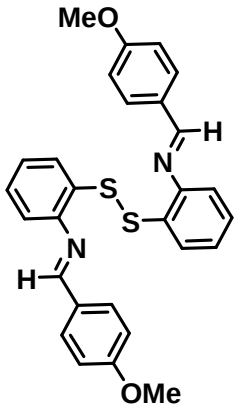
Yield: (0.579g, 93%); Yellow solid; mp 158−160 °C; IR (KBr) 1598, 1569, 1549, 1529, 1461, 1434, 1372, 1168, 946, 821, 758, 523, 463 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.04-7.07 (m, (2×1)H), 7.13-7.19 (m, (2×2)H), 7.61 (d, *J*=6.3 Hz, (2×1)H), 8.08 (d, *J*=8.4 Hz, (2×2)H), 8.27 (d, *J*=8.7 Hz, (2×2)H), 8.53 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 116.8, 123.8, 126.1, 127.0, 127.8, 129.5, 132.7, 141.0, 147.6, 149.3, 156.8; Anal. Calcd for $C_{26}H_{18}N_4O4S_2$: C, 60.69; H, 3.53; N, 10.89%. Found: C, 60.70; H, 3.54; N, 10.90%.

(15*E*)-2-(2-((*E*)-2-((pyridin-4-yl)methyleneamino)phenyl)disulfanyl)-*N*-((pyridin-4-yl)methylene)benzenamine (4c):



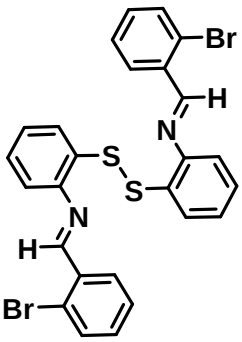
Yield: (0.380g, 89%); Grey colored solid; mp 148−150 °C; IR (KBr) 1620, 1579, 1532, 1519, 1481, 1424, 1202, 1148, 955, 823, 758, 543, 483 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.35-7.53 (m, (2×2)H), 7.83-7.89 (m, (2×3)H), 8.07 (t, *J*=8.1 Hz, (2×1)H), 8.48 (s, (2×1)H), 8.69-8.72 (m, (2×2)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 120.9, 121.6, 123.7, 125.9, 126.5, 127.4, 133.0, 140.1, 150.5, 153.7, 164.8; Anal. Calcd for $C_{24}H_{18}N_4S_2$: C, 67.58; H, 4.25; N, 13.13%. Found: C, 67.59; H, 4.26; N, 13.15%.

(15*E*)-*N*-(4-methoxybenzylidene)-2-(2-((*E*)-2-(4-methoxybenzylideneamino)phenyl)disulfanyl)benzenamine (4d):



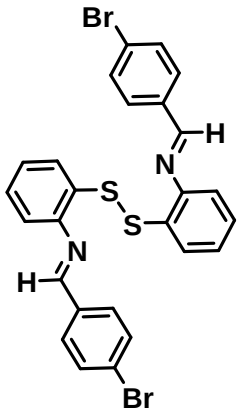
Yield: (0.436g, 90%); Light green colored solid; mp 124−126 °C; IR (KBr) 1626, 1557, 1501, 1430, 1270, 1116, 910, 764, 7-2, 583, 492 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 3.90 (s, (2×3)H), 7.04 (t, *J*=8.7 Hz, (2×3)H), 7.15-7.24 (m, (2×2)H), 7.69 (d, *J*=7.5 Hz, (2×1)H), 7.96 (d, *J*=8.1 Hz, (2×2)H), 8.44 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 55.4, 114.2, 117.1, 125.8, 126.4, 126.8, 129.1, 130.9, 131.9, 149.2, 159.1, 162.5; Anal. Calcd for $C_{28}H_{24}N_2O_2S_2$: C, 69.39; H, 4.99; N, 5.78%. Found: C, 69.41; H, 4.99; N, 5.79%.

(15*E*)-*N*-(2-bromobenzylidene)-2-(2-((*E*)-2-(2-bromobenzylideneamino)phenyl)disulfanyl)benzenamine (4e):



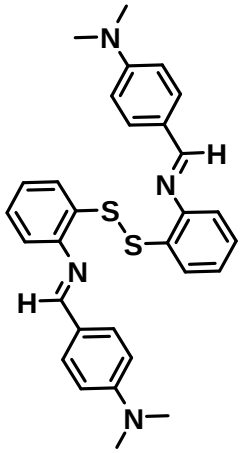
Yield: (0.518g, 89%); Grey colored solid; mp 140−142 °C; IR (KBr) 1624, 1560, 1453, 1428, 1331, 1209, 1166, 1138, 899, 766, 702, 529, 494 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.17-7.25 (m, (2×2)H), 7.37-7.47 (m, (2×3)H), 7.65-7.74 (m, (2×2)H), 8.43 (d, *J*=6.0 Hz, (2×1)H), 8.95 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 117.4, 126.1, 127.0, 127.3, 127.8, 129.6, 132.4, 132.7, 133.2, 134.3, 148.5, 158.7; Anal. Calcd for $C_{26}H_{18}Br_2N_2S_2$: C, 53.62; H, 3.12; N, 4.81%. Found: C, 53.64; H, 3.13; N, 4.83%.

(15*E*)-*N*-(4-bromobenzylidene)-2-(2-((*E*)-2-(4-bromobenzylideneamino)phenyl)disulfanyl)benzenamine (4f):



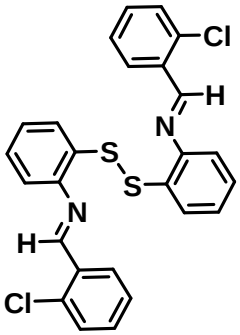
Yield: (0.530g, 91%); Grey colored solid; mp 138−140 °C; IR (KBr) 1623, 1559, 1470, 1459, 1428, 1361, 1269, 1163, 1128, 974, 756, 722, 539, 490 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.06 (dd, *J*=7.0 Hz, *J*=1.5 Hz, (2×1)H), 7.16-7.25 (m, (2×2)H), 7.60-7.70 (m, (2×3)H), 7.86 (d, *J*=8.4 Hz, (2×2)H), 8.41 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 117.1, 126.2, 126.3, 127.0, 127.1, 130.4, 132.1, 132.2, 134.9, 148.5, 158.5; Anal. Calcd for $C_{26}H_{18}Br_2N_2S_2$: C, 53.62; H, 3.12; N, 4.81%. Found: C, 53.63; H, 3.14; N, 4.82%.

(15*E*)-*N*-(4-(dimethylamino)benzylidene)-2-(2-((*E*)-2-(4-(dimethylamino)benzylideneamino)phenyl)disulfanyl)benzenamine (4g):



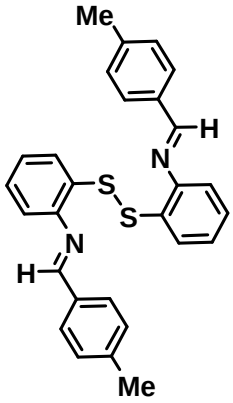
Yield: (0.470g, 92%); Yellow solid; mp 150−152 °C; IR (KBr) 1623, 1605, 1567, 1461, 1440, 1170, 1036, 810, 754, 722, 683, 492 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 3.09 (s, (2×6)H), 6.77 (d, *J*=8.1 Hz, (2×2)H), 7.04-7.22 (m, (2×3)H), 7.65 (d, *J*=7.8 Hz, (2×1)H), 7.89 (d, *J*=7.8 Hz, (2×2)H), 8.38 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 40.1, 111.5, 117.1, 124.4, 125.5, 125.8, 126.5, 130.8, 131.8, 149.7, 152.7, 159.5; Anal. Calcd for $C_{30}H_{30}N_4S_2$: C, 70.55; H, 5.92; N, 10.97%. Found: C, 70.57; H, 5.93; N, 10.99%.

(15*E*)-*N*-(2-chlorobenzylidene)-2-(2-((*E*)-2-(2-chlorobenzylideneamino)phenyl)disulfanyl)benzenamine (4h):



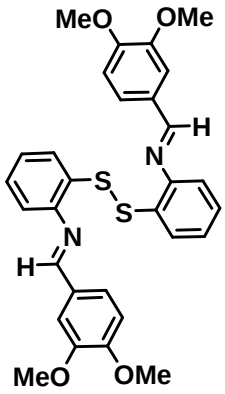
Yield: (0.439g, 89%); Grey colored solid; mp 166−168 °C; IR (KBr) 1633, 1609, 1570, 1469, 1448, 1301, 1271, 1163, 1108, 979, 743, 702, 544, 495 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.11 (d, *J*=7.7 Hz, (2×1)H), 7.16-7.25 (m, (2×2)H), 7.40-7.43 (m, (2×3)H), 7.68 (d, *J*=7.5 Hz, (2×1)H), 7.89 (dd, *J*=5.7 Hz, *J*=2.1 Hz, (2×1)H), 8.97 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 117.4, 126.1, 127.1, 127.3, 127.4, 129.2, 129.9, 130.0, 132.5, 133.1, 136.1, 136.2, 148.7, 156.5; Anal. Calcd for $C_{26}H_{18}Cl_2N_2S_2$: C, 63.28; H, 3.68; N, 5.68%. Found: C, 63.30; H, 3.69; N, 5.70%.

(15*E*)-*N*-(4-methylbenzylidene)-2-(2-((*E*)-2-(4-methylbenzylideneamino)phenyl)disulfanyl)benzenamine (4i):



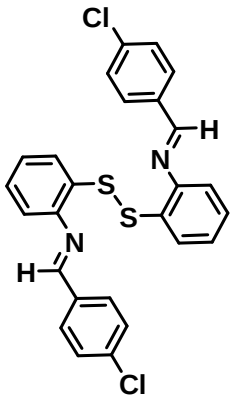
Yield: (0.412g, 91%); Grey colored solid; mp 120−122 °C; IR (KBr) 1622, 1598, 1557, 1468, 1340, 1178, 1106, 910, 764, 722, 563, 495 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 2.47 (s, (2×3)H), 7.08 (d, *J*=7.2 Hz, (2×1)H), 7.15-7.23 (m, (2×1)H), 7.32-7.37 (m, (2×2)H), 7.72 (d, *J*=7.5 Hz, (2×1)H), 7.82 (t, *J*=7.8 Hz, (2×1)H), 7.92 (d, *J*=7.5 Hz, (2×2)H), 8.48 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 21.6, 117.1, 125.9, 126.6, 126.8, 129.4, 129.5, 129.6, 129.7, 131.9, 133.5, 142.2, 149.0, 159.8; Anal. Calcd for $C_{28}H_{24}N_2S_2$: C, 74.30; H, 5.34; N, 6.19%. Found: C, 74.32; H, 5.35; N, 6.20%.

(15*E*)-N-(3,4-dimethoxybenzylidene)-2-(2-((*E*)-2-(3,4-dimethoxybenzylideneamino)phenyl)disulfanyl)benzenamine (4j):



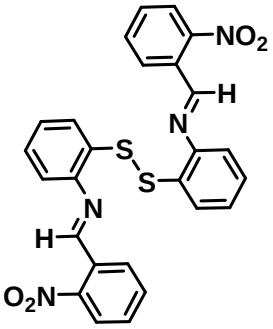
Yield: (0.458g, 84%); Light green colored solid; mp 130−132 °C; IR (KBr) 1622, 1600, 1577, 1492, 1320, 1270, 1136, 940, 854, 722, 663, 490 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 3.84 (s, (2×6)H), 6.81-6.86 (m, (2×1)H), 6.97 (dd, *J*=7.4 Hz, *J*=1.2 Hz, (2×1)H), 7.06-7.17 (m, (2×2)H), 7.27-7.35 (m, (2×2)H), 7.66 (s, (2×1)H), 8.31 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 55.5, 55.6, 109.2, 110.3, 116.9, 124.3, 125.5, 126.2, 126.6, 129.0, 131.5, 148.7, 149.1, 152.0, 159.0; Anal. Calcd for $C_{30}H_{28}N_2O_4S_2$: C, 66.15; H, 5.18; N, 5.14%. Found: C, 66.17; H, 5.19; N, 5.16%.

(15*E*)-*N*-(4-chlorobenzylidene)-2-(2-((*E*)-2-(4-chlorobenzylideneamino)phenyl)disulfanyl)benzenamine (4k):



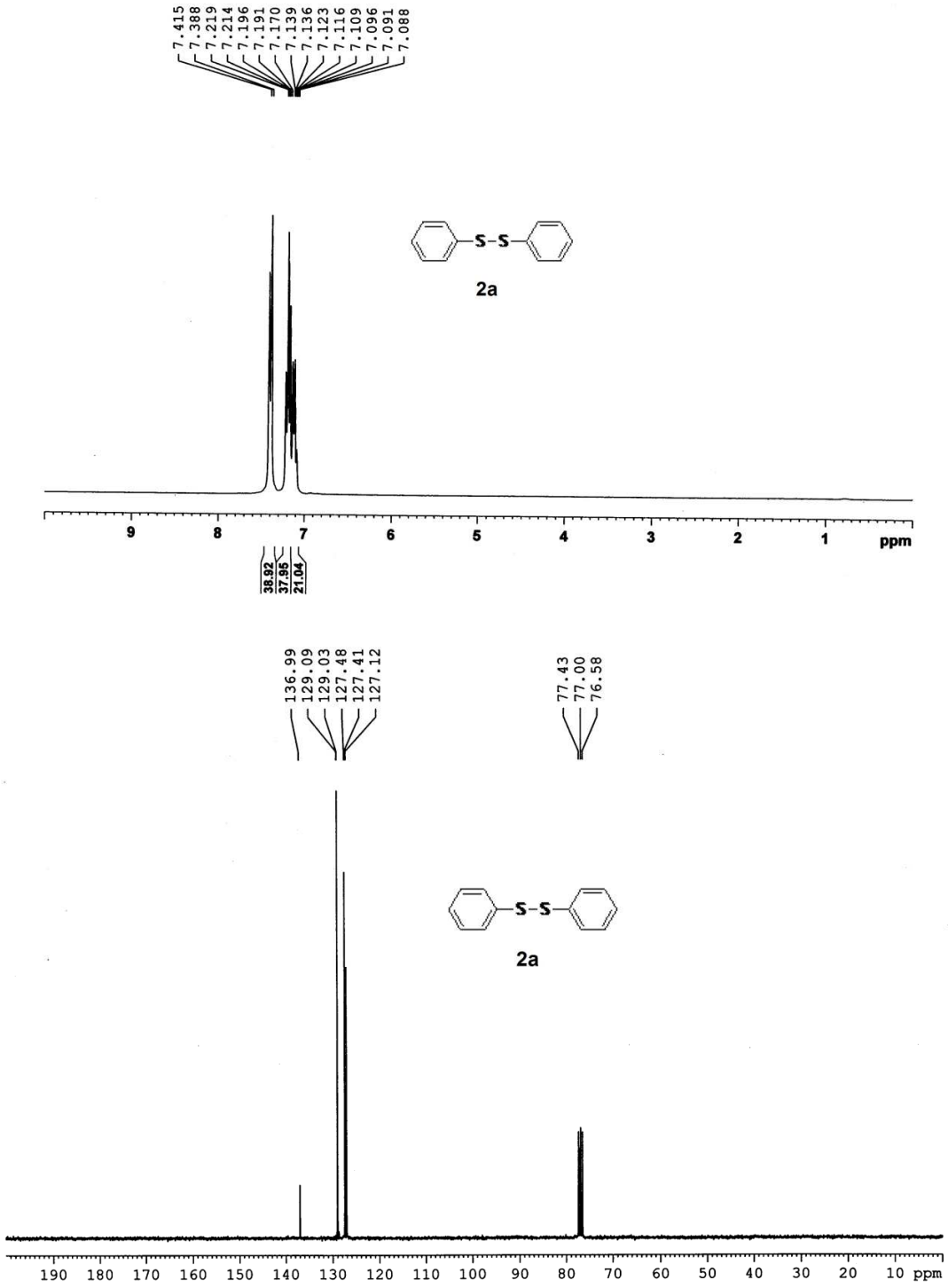
Yield: (0.444g, 90%); Grey colored solid; mp 149−151 °C; IR (KBr) 1623, 1599, 1560, 1419, 1348, 1302, 1257, 1153, 1108, 989, 753, 722, 534, 493 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.07-7.09 (m, (2×1)H), 7.19-7.27 (m, (2×4)H), 7.33-7.36 (m, (2×1)H), 7.54-7.60 (m, (2×2)H), 8.47 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 117.2, 126.16, 126.22, 126.9, 127.1, 128.3, 130.1, 130.2, 134.9, 149.2, 158.4; Anal. Calcd for $C_{26}H_{18}Cl_2N_2S_2$: C, 63.28; H, 3.68; N, 5.68%. Found: C, 63.30; H, 3.69; N, 5.69%.

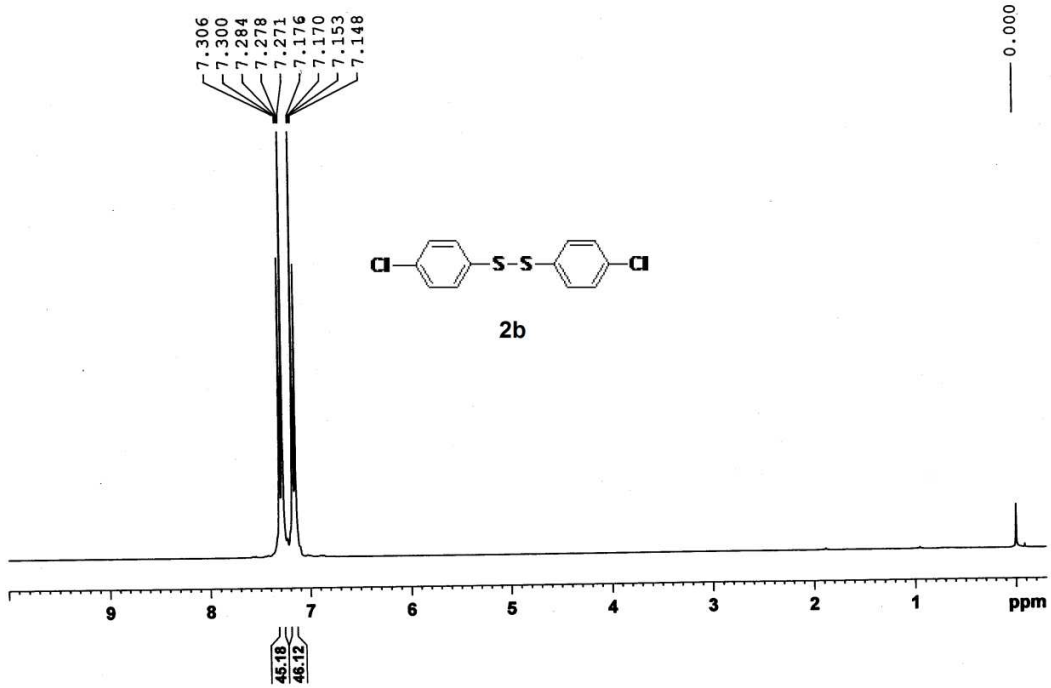
(15*E*)-*N*-(2-nitrobenzylidene)-2-(2-((*E*)-2-(2-nitrobenzylideneamino)phenyl)disulfanyl)benzenamine (4l):

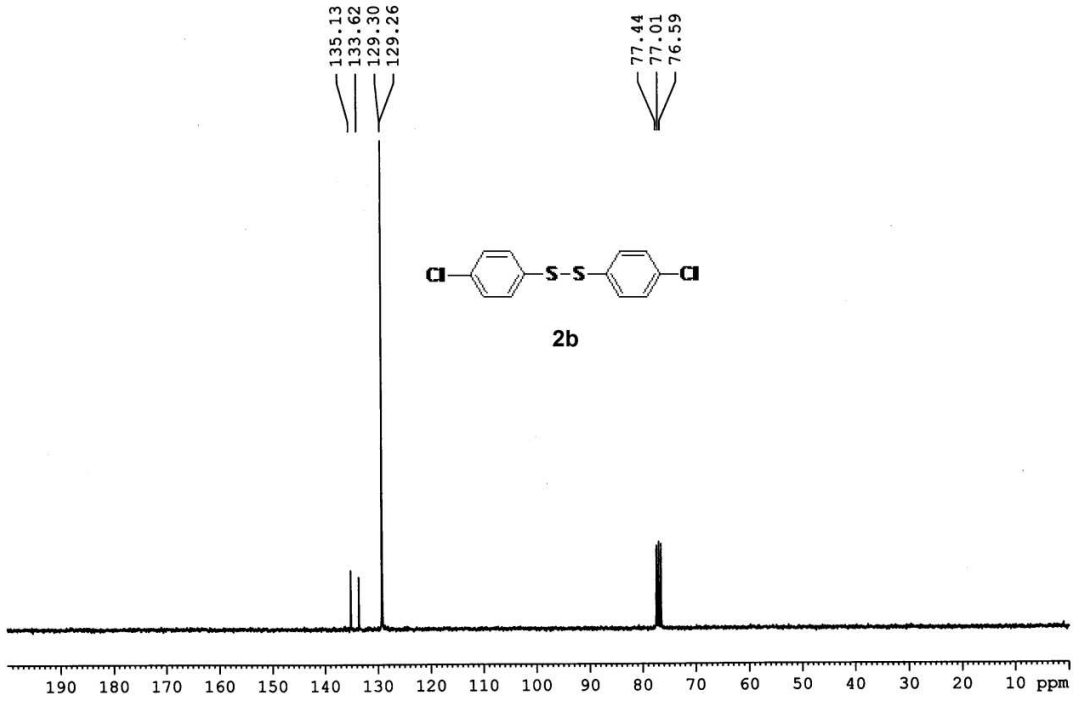


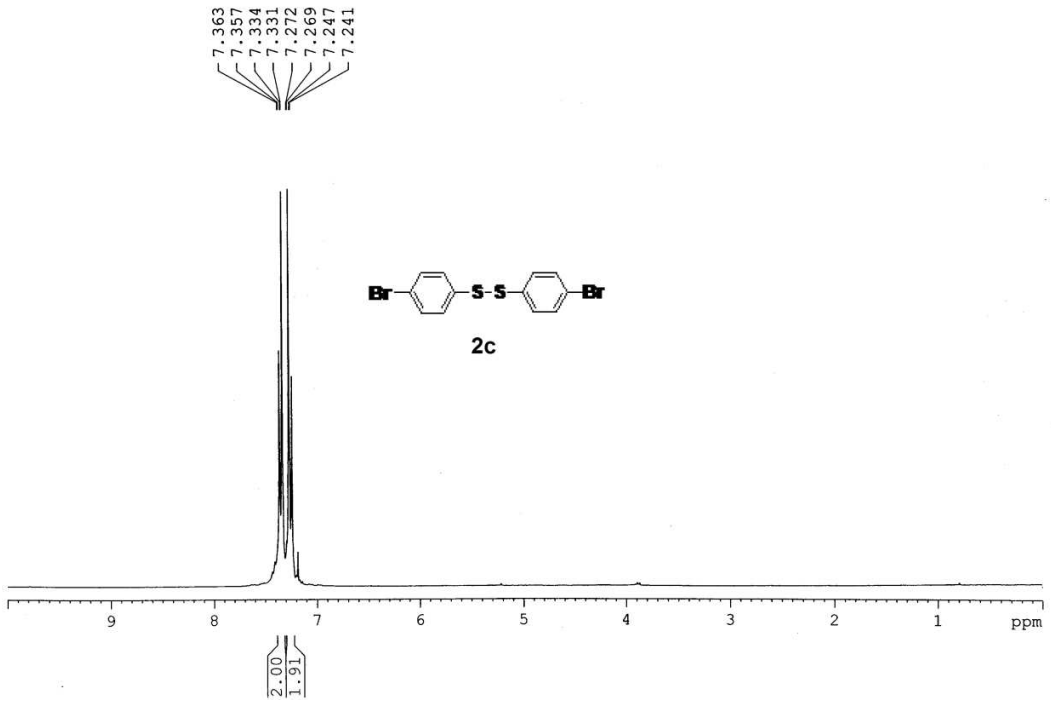
Yield: (0.473g, 92%); Yellow solid; mp 162−164 °C; IR (KBr) 1608, 1589, 1551, 1509, 1477, 1424, 1362, 1268, 956, 821, 766, 536, 473 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$; Me_4Si) δ 7.16-7.28 (m, (2×3)H), 7.63-7.70 (m, (2×2)H), 7.78 (t, *J*=7.5 Hz, (2×1)H), 8.10 (d, *J*=7.8 Hz, (2×1)H), 8.46 (d, *J*=7.5 Hz, (2×1)H), 9.01 (s, (2×1)H); ^{13}C NMR (75 MHz, $CDCl_3$; Me_4Si) δ 117.6, 124.5, 126.3, 127.3, 127.8, 130.2, 130.9, 131.5, 132.4, 133.7, 148.2, 149.3, 155.6; Anal. Calcd for $C_{26}H_{18}N_4O_4S_2$: C, 60.69; H, 3.53; N, 10.89%. Found: C, 60.71; H, 3.55; N, 10.90%.

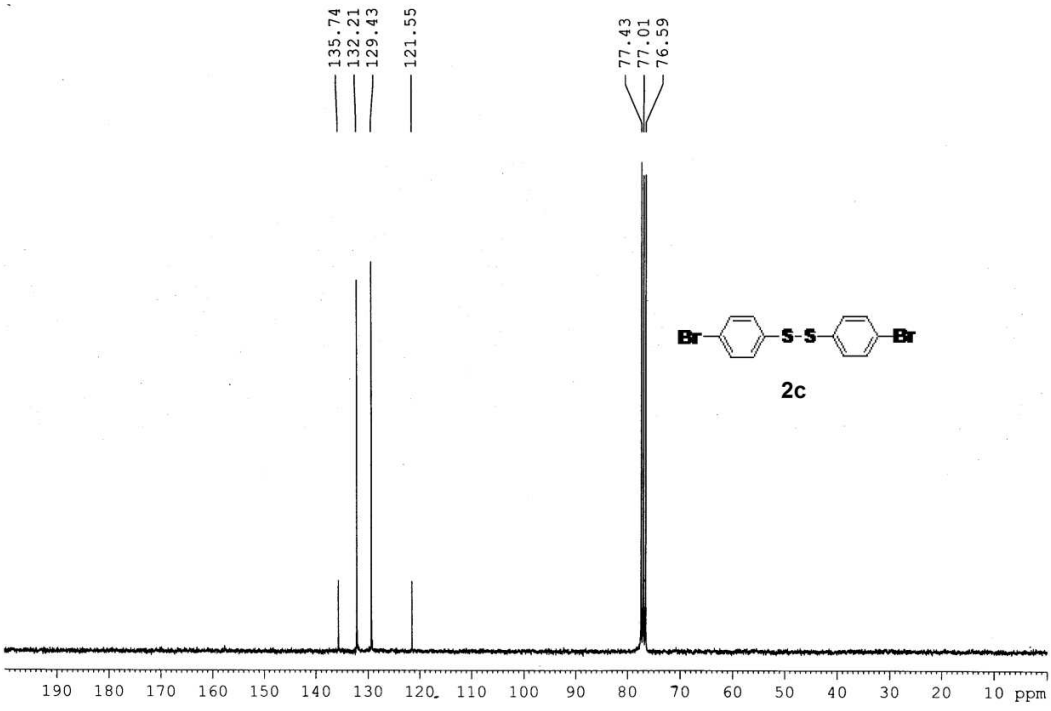
Spectral Data of Didulphides 2a-2l

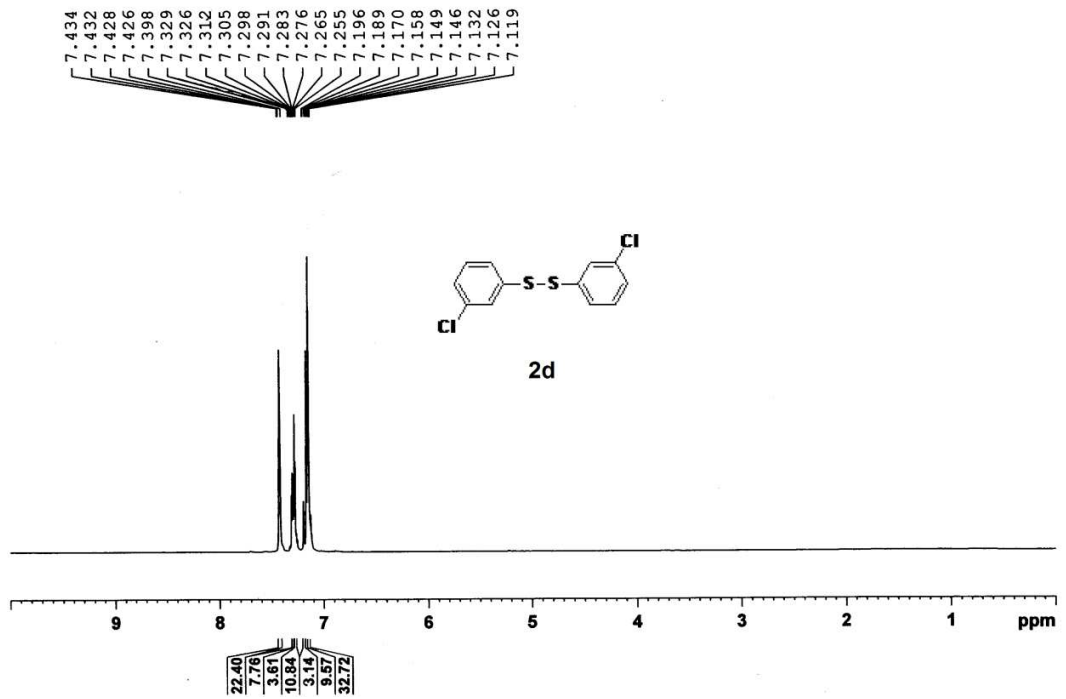


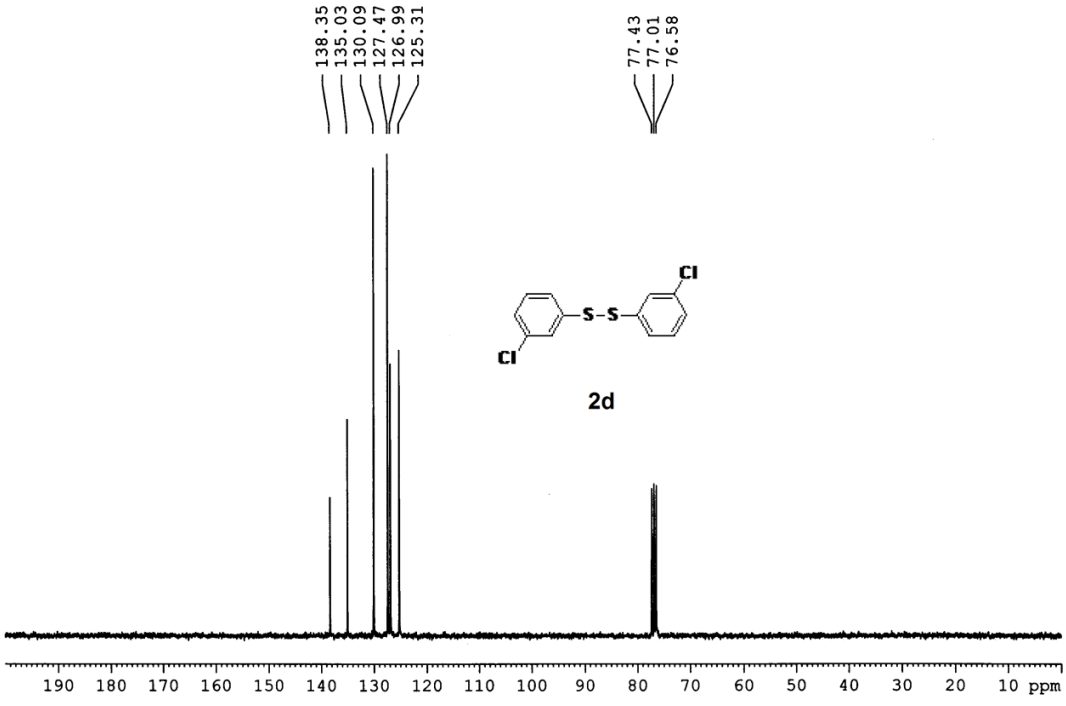

7.306
7.300
7.284
7.278
7.271
7.176
7.170
7.153
7.148
0.000
Cl
S-S
Cl
2b
9
8
7
6
5
4
3
2
1
ppm
45.18
46.12

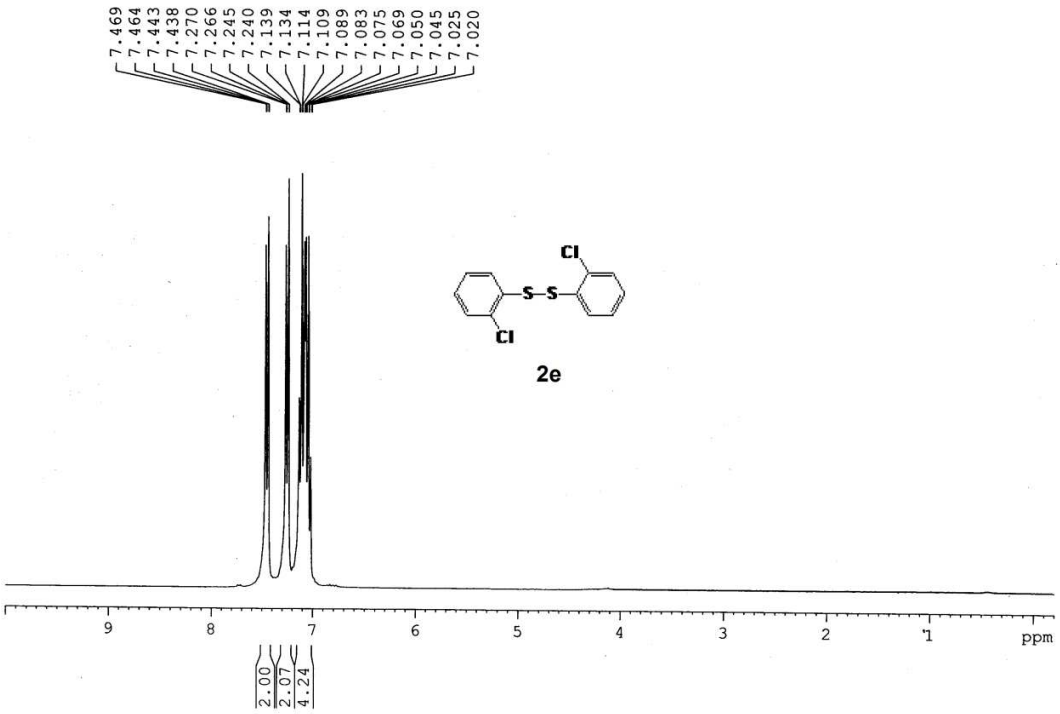

135.13
133.62
129.30
129.26
77.44
77.01
76.59
Cl
S-S
Cl
2b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.363
7.357
7.334
7.331
7.272
7.269
7.247
7.241
Br
S-S
Br
2c
9
8
7
6
5
4
3
2
1
ppm
2.00
1.91


135.74
132.21
129.43
121.55
77.43
77.01
76.59
Br
S-S
Br
2c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10 ppm


7.434
7.432
7.428
7.426
7.398
7.329
7.326
7.312
7.305
7.298
7.291
7.283
7.276
7.265
7.255
7.196
7.189
7.170
7.158
7.149
7.146
7.132
7.126
7.119
Cl
S-S
Cl
2d
9
8
7
6
5
4
3
2
1
ppm
22.40
7.76
3.61
10.84
3.14
9.57
32.72


138.35
135.03
130.09
127.47
126.99
125.31
77.43
77.01
76.58
Cl
S-S
Cl
2d
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10 ppm


7.469
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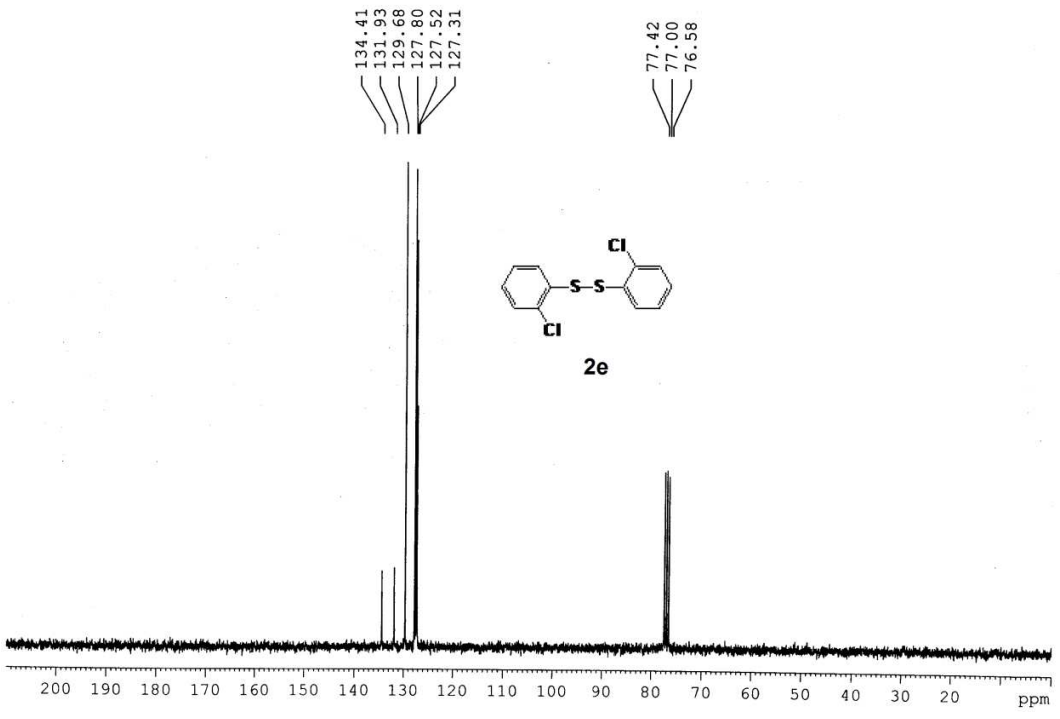
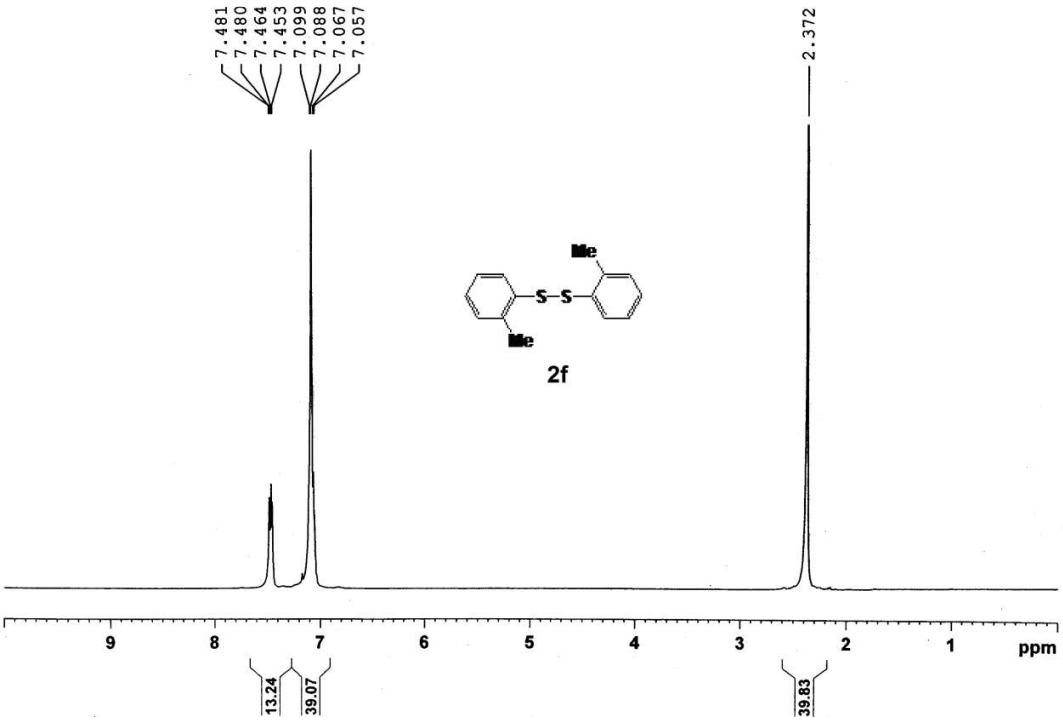
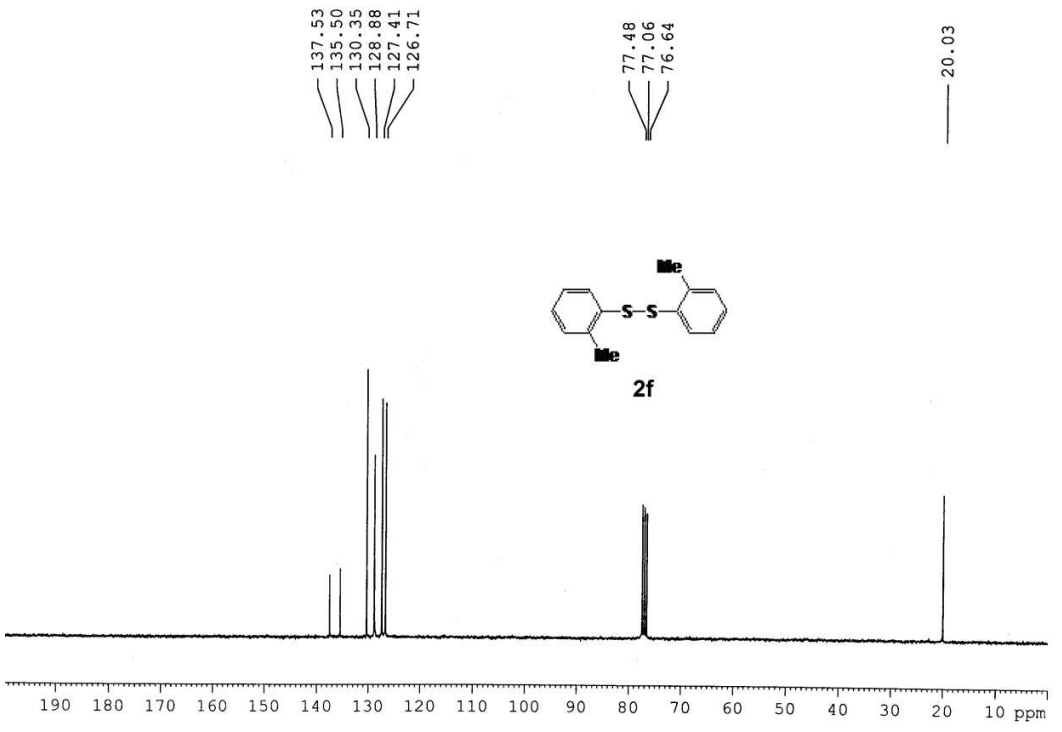
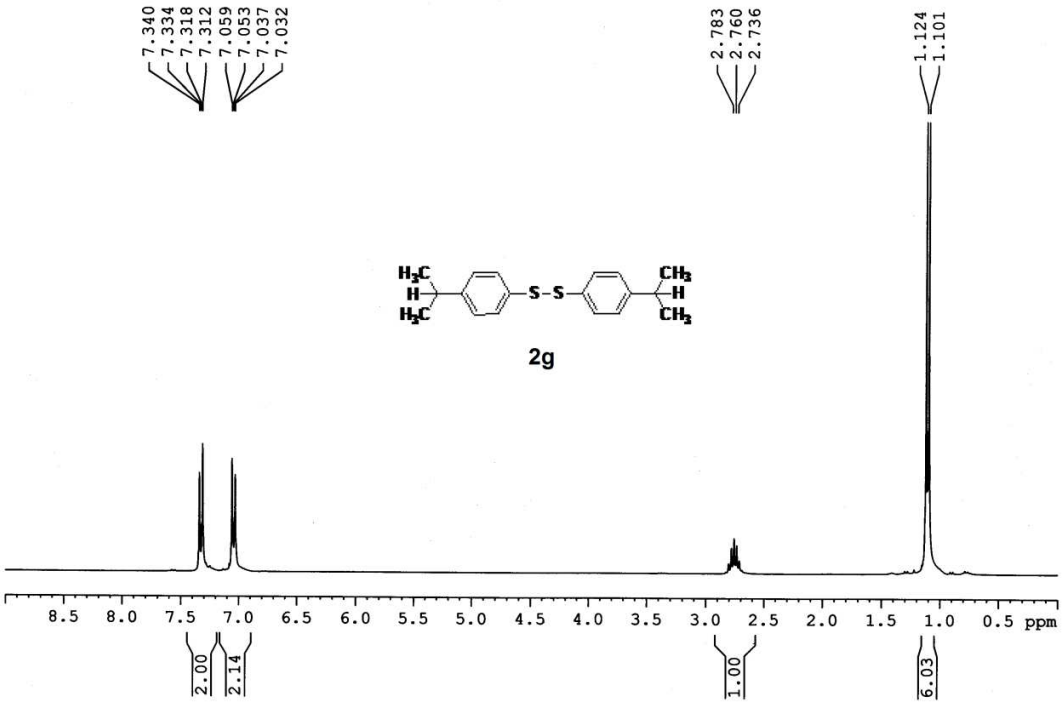
7.443
7.438
7.270
7.266
7.245
7.240
7.139
7.134
7.114
7.109
7.089
7.083
7.075
7.069
7.050
7.045
7.025
7.020
Cl
S–S
Cl
2e
9
8
7
6
5
4
3
2
1
ppm
2.00
2.07
4.24


134.41
131.93
129.68
127.80
127.52
127.31
77.42
77.00
76.58
Cl
S–S
Cl
2e
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
ppm


7.481
7.480
7.464
7.453
7.099
7.088
7.067
7.057
2.372
Me
S–S
Me
2f
9
8
7
6
5
4
3
2
1
ppm
13.24
39.07
39.83


137.53
135.50
130.35
128.88
127.41
126.71
77.48
77.06
76.64
20.03
Me
S–S
Me
2f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10 ppm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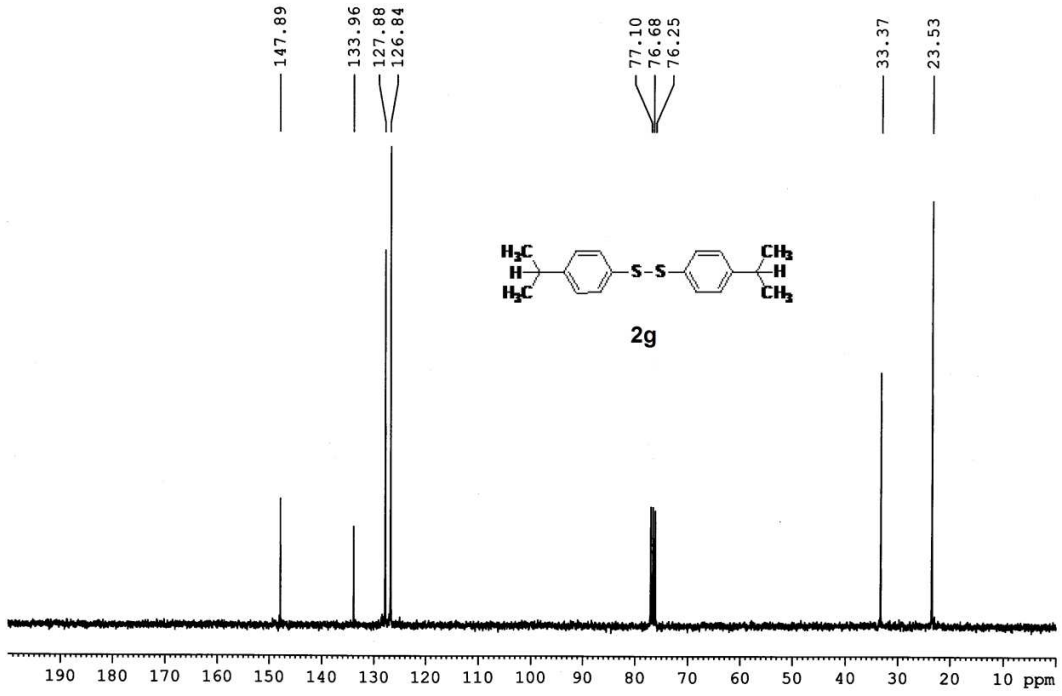
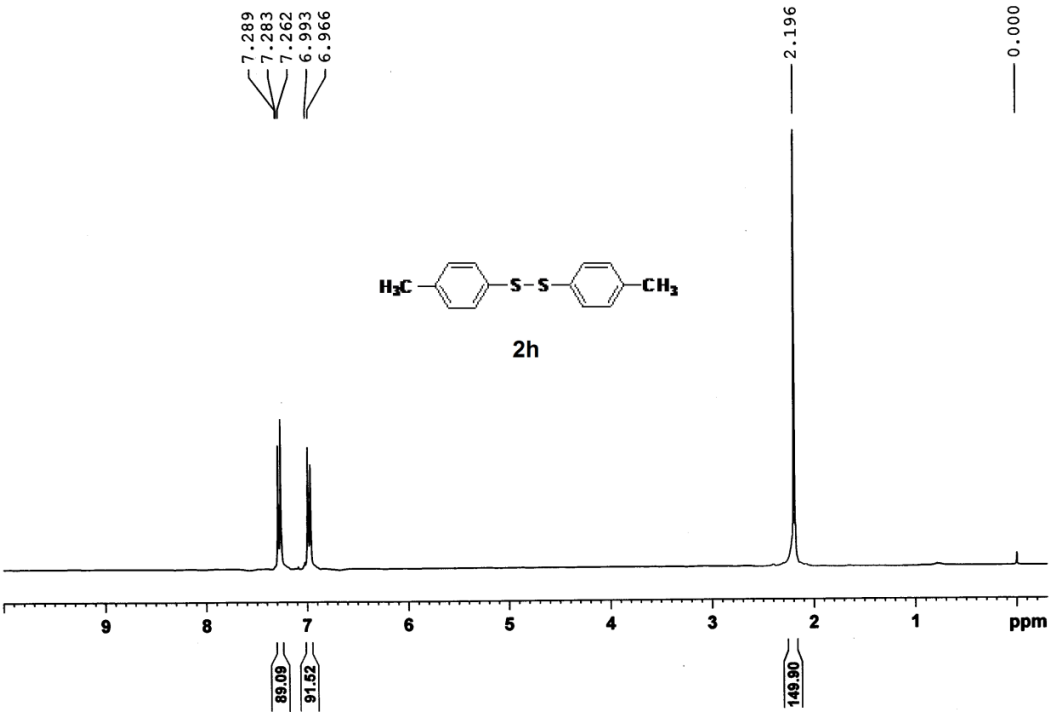
7.334
7.318
7.312
7.059
7.053
7.037
7.032
2.783
2.760
2.736
1.124
1.101
2g
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
2.14
1.00
6.03


147.89
133.96
127.88
126.84
77.10
76.68
76.25
33.37
23.53
2g
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.289
7.283
7.262
6.993
6.966
2.196
0.000
H3C
S-S
CH3
2h
9
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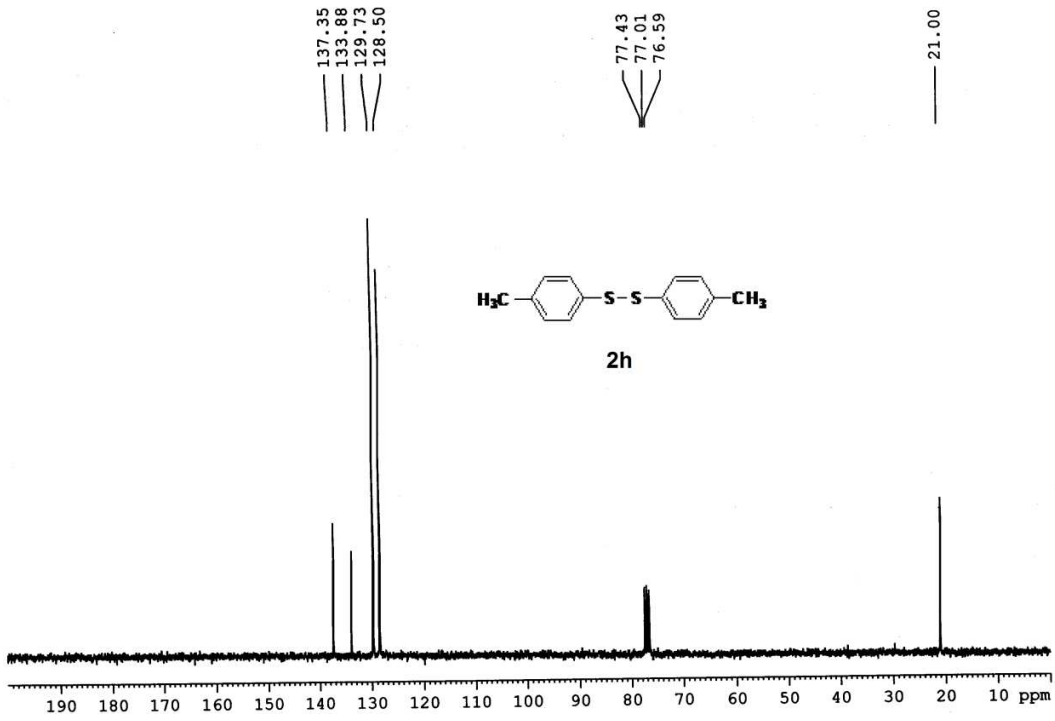
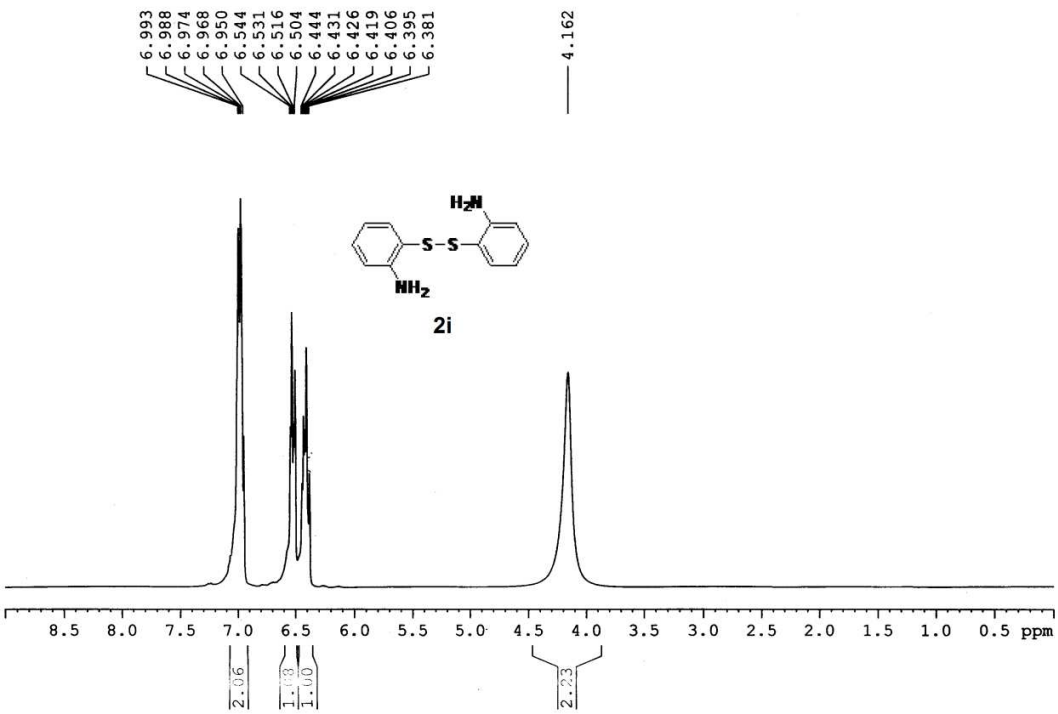
4
3
2
1
ppm
89.09
91.52
149.90


137.35
133.88
129.73
128.50
77.43
77.01
76.59
21.00
H3C
S-S
CH3
2h
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10 ppm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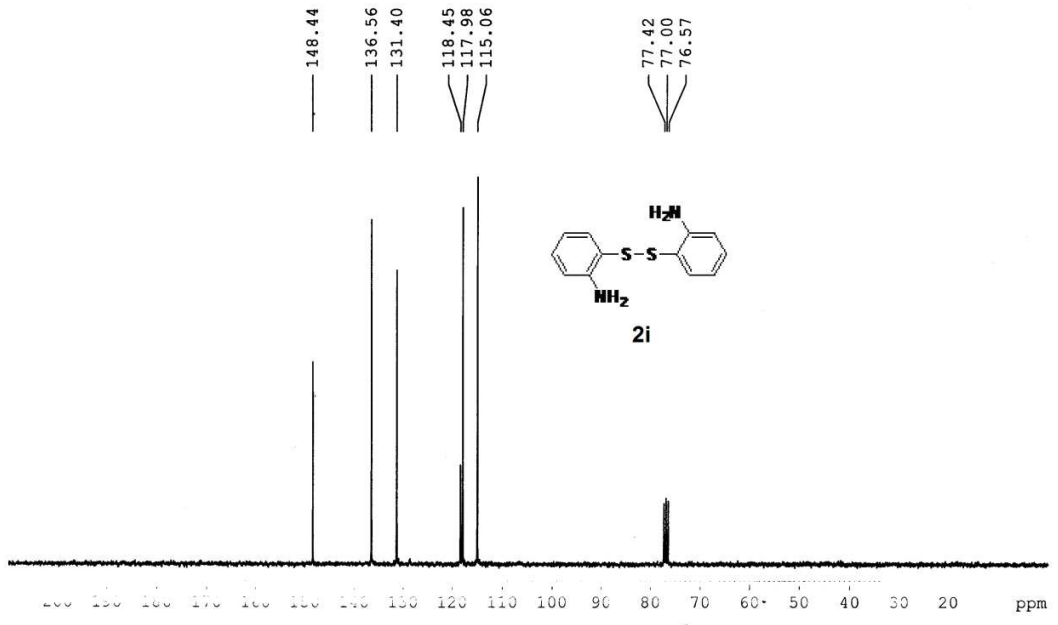
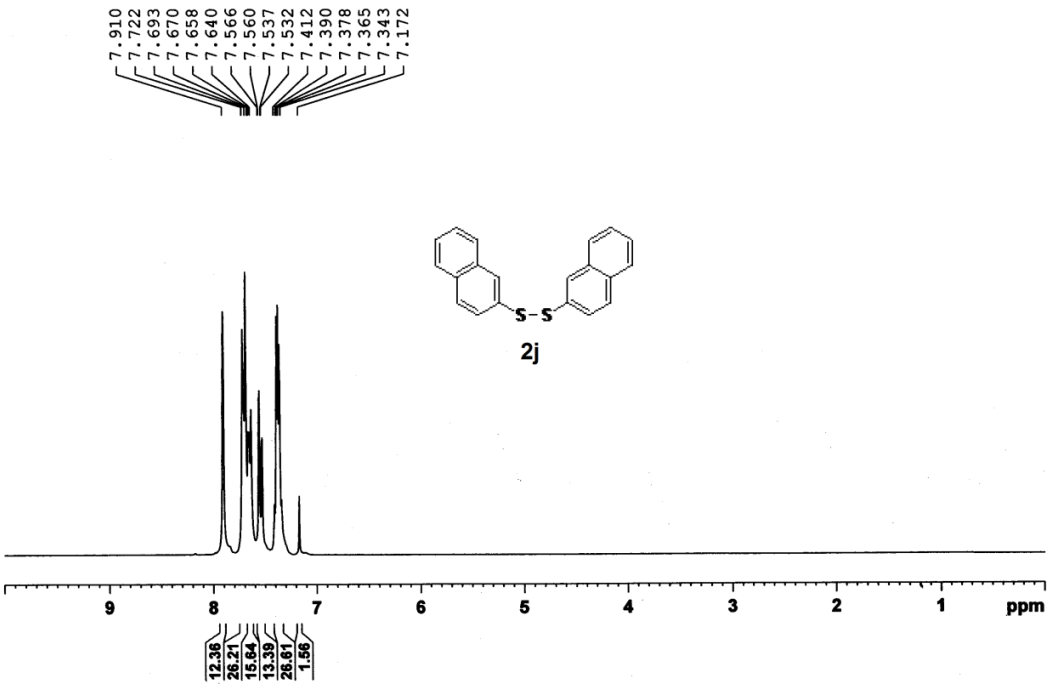
6.988
6.974
6.968
6.950
6.544
6.531
6.516
6.504
6.444
6.431
6.426
6.419
6.406
6.395
6.381
4.162
H2N
S-S
NH2
2i
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.06
1.08
1.00
2.23


148.44
136.56
131.40
118.45
117.98
115.06
77.42
77.00
76.57
H2N
S-S
NH2
2i
ppm


7.910
7.722
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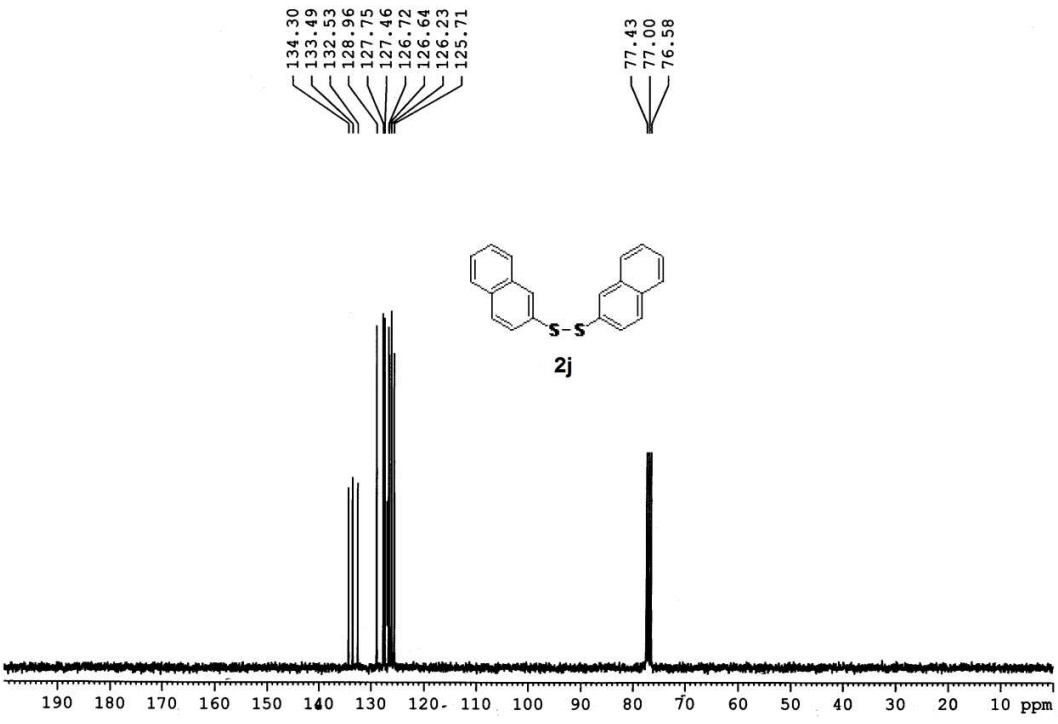
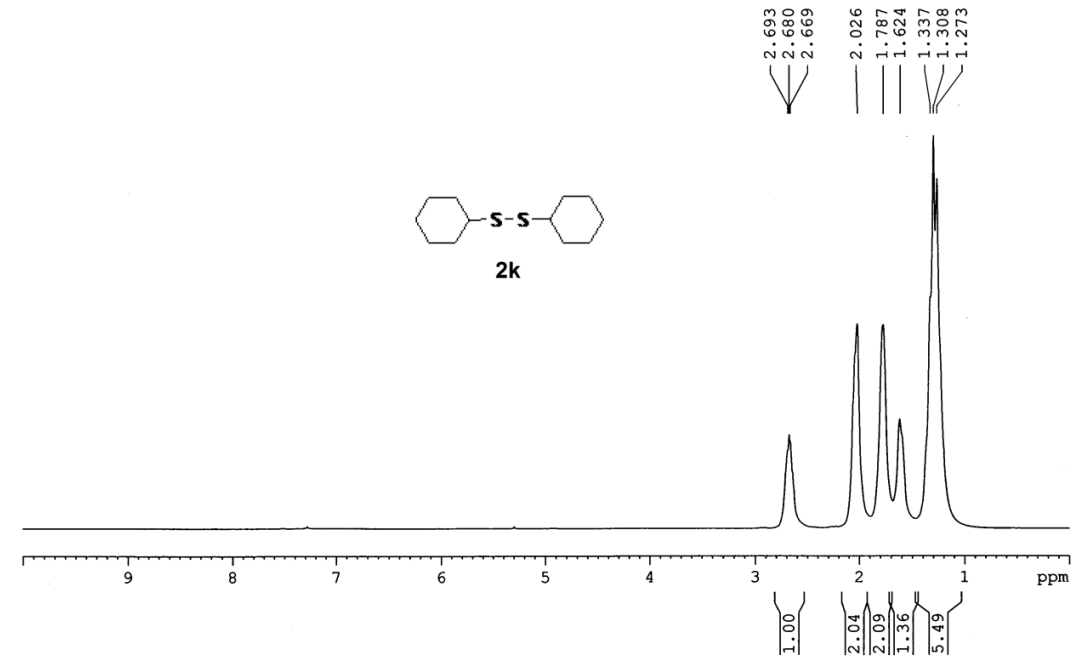
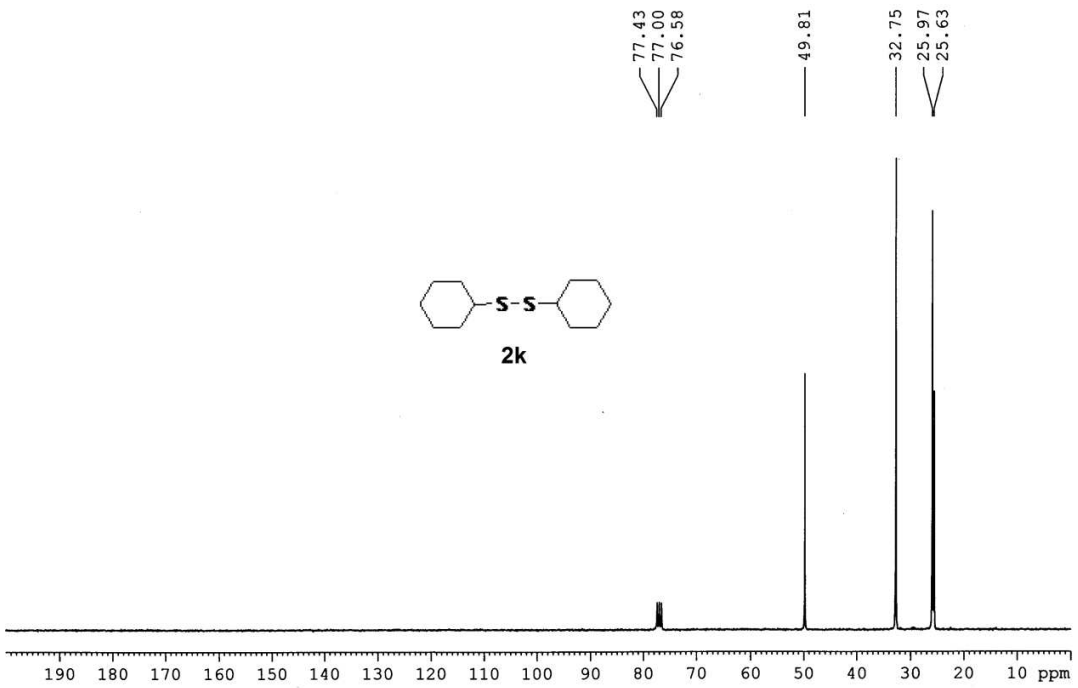
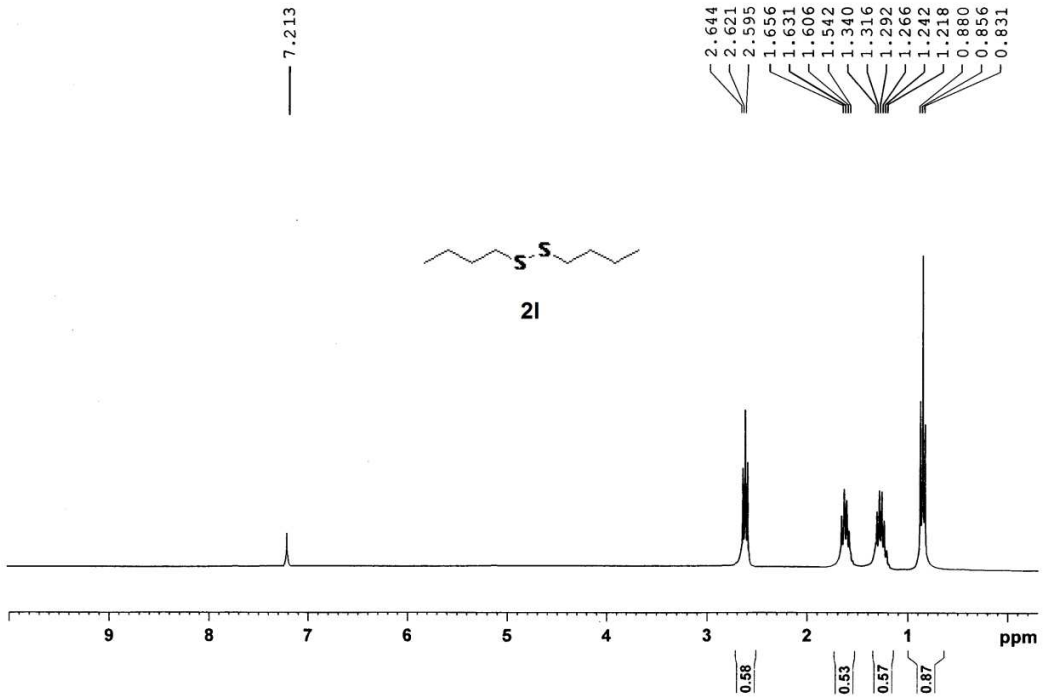
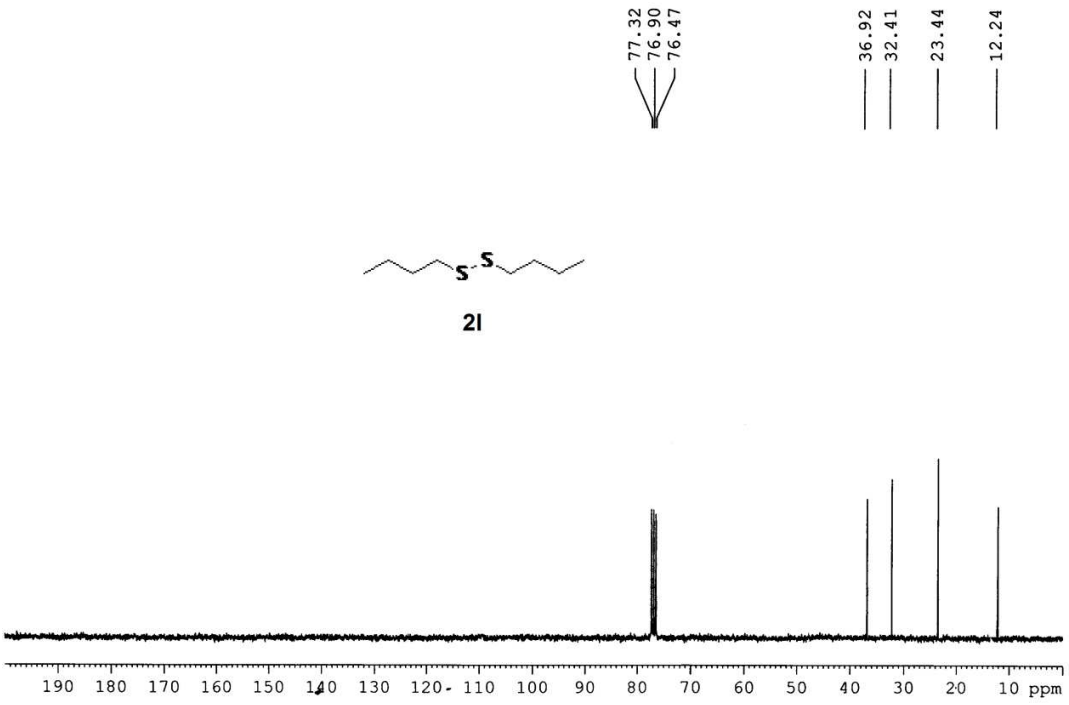
7.670
7.658
7.640
7.566
7.560
7.537
7.532
7.412
7.390
7.378
7.365
7.343
7.172
S-S
2j
9
8
7
6
5
4
3
2
1
ppm
12.36
26.21
15.64
13.39
26.61
1.56


134.30
133.49
132.53
128.96
127.75
127.46
126.72
126.64
126.23
125.71
77.43
77.00
76.58
S-S
2j
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10 ppm


2.693
2.680
2.669
2.026
1.787
1.624
1.337
1.308
1.273
S-S
2k
9
8
7
6
5
4
3
2
1
ppm
1.00
2.04
2.09
1.36
5.49


77.43
77.00
76.58
49.81
32.75
25.97
25.63
S-S
2k
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.213
2.644
2.621
2.595
1.656
1.631
1.606
1.542
1.340
1.316
1.292
1.266
1.242
1.218
0.880
0.856
0.831
2l
9
8
7
6
5
4
3
2
1
ppm
0.58
0.53
0.57
0.87


77.32
76.90
76.47
36.92
32.41
23.44
12.24
2l
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10 ppm